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New Techniques to Evaluate the Incendiary Behavior of Insulators

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Abstract – New techniques for evaluating the incendiary behavior of insulators is presented. The onset of incensive brush discharges in air is evaluated using standard spark probe techniques for the case simulating approaches of an electrically grounded sphere to a charged insulator in the presence of a flammable atmosphere. However, this standard technique is unsuitable for the case of brush discharges that may occur during the charging-separation process for two insulator materials. We present experimental techniques to evaluate this hazard in the presence of a flammable atmosphere which is ideally suited to measure the incendiary nature of micro-discharges upon separation, a measurement never before performed. Other measurement techniques unique to this study include; surface potential measurements of insulators before, during and after contact and separation, as well as methods to verify fieldmeter calibrations using a charge insulator surface opposed to standard high voltage plates.

Key words: Kapton® polyimide film, incendiary discharges, brush discharges, contact and frictional electrification, ignition hazards, insulators, contact angle, surface potential measurements.

1.0 Background

Standard techniques used to evaluate the incendiary nature of materials such as the Spark Incendivity Test [1], are successful in determining the likelihood of incensive brush discharges caused by a grounded object (most likely a human finger) in contact with a charged insulator surface. Although such techniques are widely used throughout Europe and lately the United States, they are not successful in determining the incendiary nature of discharges between two insulator materials upon immediate separation. The contact and separation of insulator materials can result into two types of discharges. The first type results from the excessive amounts of charge during separation which commonly produces electric fields above the breakdown of air. These micro-discharges are sometimes referred to as back discharges [2] are rarely discussed in the literature but are thought to be due to field emission in the case of metals [3] and air breakdown in the case of insulators. As the materials separate further, the micro-discharges result in a surface charge equilibrium which produces a fixed amount of charge on each insulator. It is normally at this point at which surface potentials are

measured using standard fieldmeter techniques. To test for incendivity, a grounded sphere approaches the charged surface and the convergence of the electric field forces another gas breakdown in the form of a common brush discharge which occurs at an even later point in time. Although the evaluation of the incendiary nature of brush discharges is well determined by probe techniques, it is not clear whether the previous discharges that occurred during the separation process are incendive. The authors can see no reason why these micro-discharges can be labeled as non-incendive a priori without experimentation.

The aim of this paper is to convey new techniques used to evaluate the incendiary nature of insulator-insulator contact charging and the resulting separation. As an example we used the famous dielectric polyimide (5 mil) Kapton® HN by DuPont™. A background on the electrostatic properties of Kapton® will be provided including surface and volume resistivity measurements, charge decay measurements and contact angle measurements followed by conventional Spark Incendivity tests. Proper methods to measure the voltage necessary at which the onset of brush discharges occurs will be presented along with unique fieldmeter calibration results. A new method to measure surface potentials before, during and after separation will also be presented. Finally, insulator-insulator incendiary tests will be discussed along with additional Spark Incendivity testing.

2.0 Initial Tests

2.1 Surface and Volume Resistivity Measurements

The first series of tests performed to evaluate the electrostatic properties of a material is surface resistance. Surface resistance measurements are the main test method used in industry to characterize the ESD properties of materials, since it is believed that charge deposited onto the surface of a material will “leave” (or decay) easier from a material with lower surface resistance than from a material with high surface resistance. Surface resistance is the ratio of the DC voltage to the current flowing between two electrodes of specified configuration that contact the same side of the material and is expressed in ohms (Ω). The surface resistance tests are performed per the requirements of the ESD Association Standard Test Method ESD STM11.11 [4]. These measurements are taken using a PRS-801 resistance system with an Electro Tech System (ETS) PRF-911 concentric ring resistance probe (Cal # M81019). The tests require a five pound weight on top of cylindrical electrodes and were conducted at both ambient and low humidity conditions. Materials with a surface resistance less than $10^4 \Omega$ are considered conductive. Materials between $10^4 \Omega$ and $10^{11} \Omega$ are statically dissipative and materials with a surface resistance above $10^{11} \Omega$ are insulating according to ANSI/ESD standards.

Volume resistivity tests are also conducted to measure conductivity through the material as opposed to conductivity along the surface. These tests are conducted using the same PRS-801 resistance system with the Electro Tech System PRF-911 concentric ring resistance probe but are performed in accordance with ESD Association Standard Test Method ESD STM11.12 [5].

2.1.1 Surface and Volume Resistivity Results

The data below in Table 1 is from five or six samples of 5" × 5" size cut from the polyimide roll and tested per ESD STM11.11-2001 at a variety of relative humidities (RH) and at 23 ± 3° C. Three humidities were chosen to simulate extreme conditions at which the material could be exposed to such as warm summer months (~70% RH), normal ambient conditions (~50% RH) and during drier winter months (0% RH).

The results indicate surface resistances and volume resistivities greater than $10^{11} \Omega$ (or $\Omega \text{ cm}$) in both cases indicating that the Kapton® is highly resistive and should act as a good insulator. Although there appears to be a slight drop in resistivity at 70% RH, charge should not be expected to dissipate along the surface or through this material due to the high values. Resistance measurements alone imply that it may not be possible to electrically ground this material.

Resistance measurements are commonly performed on materials because it is assumed that test materials behave similar to electric circuits which dissipates electric charge exponentially in time through a well defined time constant. Using this simplification, any measurement of resistance is directly proportional to the time constant $\tau = RC$ with only capacitance as the unknown. In real materials however, charge decay properties rarely exhibit pure exponential behavior thus a better method to determine the electrostatic dissipative properties of a material is through charge decay measurements directly.

Table 1. Surface and Volume Resistivity Results for Kapton® at various humidities.

	Surface Resistance	Volume Resistivity
Kapton® (0% RH)	21.4 ± 10.8 TΩ	814 ± 128 TΩ cm
Kapton® (50% RH)	8.23 ± 3.67 TΩ	1301 ± 900 TΩ cm
Kapton® (70% RH)	2.74 ± 1.62 TΩ	569 ± 128 GΩ cm

(Note: MΩ is mega ohms $10^6 \Omega$, GΩ is giga ohms $10^9 \Omega$, and TΩ is tera ohms $10^{12} \Omega$)

2.2 Charge Decay Measurements

Charge Decay tests are performed on candidate materials to test their ability to dissipate charge. Corona charge deposition is chosen because surface charge levels can be deposited in a controlled manner. Tests are performed using a JCI 155v5 Charge Decay Test Unit conforming to British Standard 7506 [6]. This device deposits a consistent amount of positive or negative charge onto the surface of a material by ionizing the air molecules with high voltage corona needle points up to ±10,000 volts. The JCI 155v5 sits on top of a JCI 176 Charge Measurement Sample Support system which has the ability to measure the total amount of charge transferred to the sample (see Figure 1). The total amount of charge transferred to the sample consists of both the charge that “leaves” the surface of the sample and the charge that remains. The samples are mounted between two conducting isolated (ungrounded) plates. Once the charge is deposited onto the surface of the test material, the plate containing the corona points is retracted (within 20 milliseconds) and a fieldmeter is exposed. This fieldmeter measures and records the surface voltage on the material as a function of time. The time it takes for the surface voltage to reach 1/e (1/2.71828) or 37% of

its maximum value is called the *decay time* or τ . For materials to possess “good” ESD behavior, they should dissipate the charge faster than one could deposit it in the field. The test standard specifies that typical decay times should be on the order of ~ 1 second but it notes that this also depends upon the charge generating mechanism.

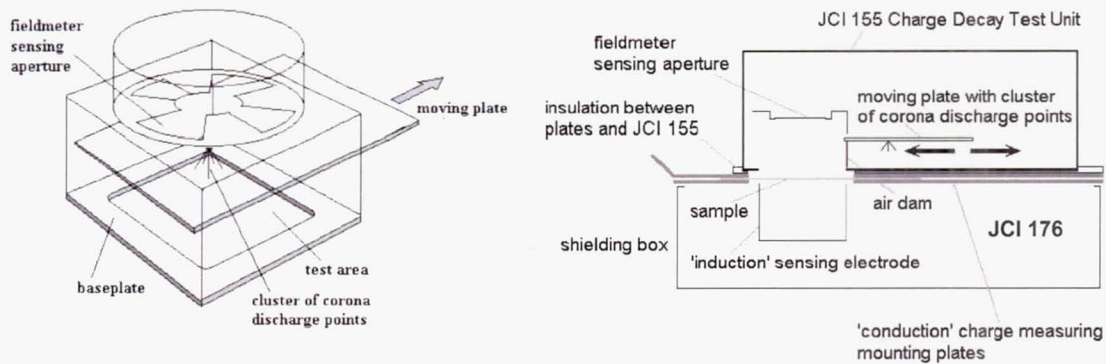


Figure 1. Schematic of the JCI 155v5 Charge Decay Test Unit and JCI 176 Sample Support.

A number of charge decay tests were performed on the Kapton® samples for comparison. The initial surface potential readings on all of the samples is about -1.8 kV based on the applied voltage and allowed charging time which is on the order of 20 milliseconds. The charging area is also quite small on the order of 1-2 square inches. After the corona is applied, the surface potential is monitored until it drops below 10% of its initial value which depends on its charge decay properties. The strongest influence on these properties is the environmental relative humidity (RH). To acclimate to these conditions, the test samples and instrumentation are usually placed in an environmental chamber for a minimum of 24 hours. Multiple tests were performed on the polyimide film at various relative humidities whose results are shown in Figure 2.

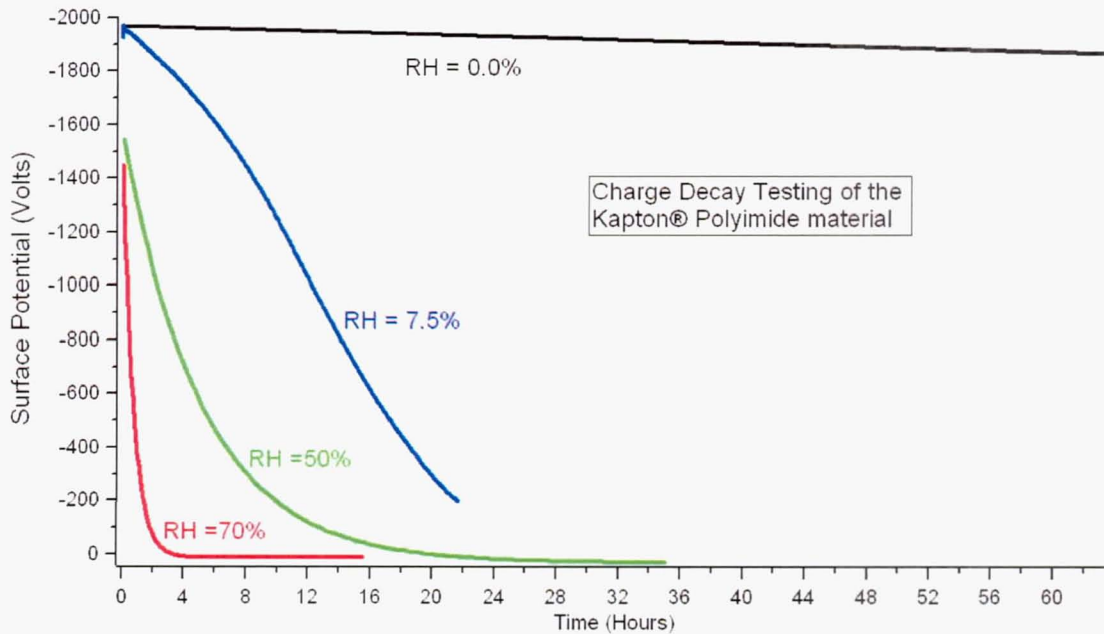


Figure 2. Charge decay measurements for Kapton® polyimide film under various humidity conditions.

2.2.1 Charge Decay Results

It is clear from the preliminary charge decay tests that Kapton® does not charge decay in less than one second regardless of humidity. However, the charge decay results above indicate that is indeed possible for charge to dissipate from Kapton provided ample time has passed. For example, at high humidities at or above 70% RH charge can dissipate from the surface in about 42 minutes according to Figure 2, while moderate humidities ~50% RH require longer charge decay times. For 50% RH the decay time was 5.12 hours while at 7.5% RH the decay time was about 16 hours. At very low humidities (near 0% RH) charge is not expected to decay in less than 446 hours or 18 ½ days. All of the above assumes that charge has a well-defined path to ground. Although the material fails to dissipate charge fast enough to pass the corona charge decay test standard, the graph does indicate that if the proper connection to ground it is possible for charge to dissipate whose rate is strongly dependent upon humidity. Clearly, humidity plays a role in charge decay characteristics.

2.3 Contact Angle Measurements

A common method to measure a material's ability to adsorb water is through contact angle measurements. A VCA Optima Surface Analysis System was used for contact angle measurements. The system is enclosed in an environmental chamber so that the humidity can be controlled to within +/- 5% RH. Droplets of de-ionized water (17.8 M-ohm) were generated using a Hamilton syringe. The droplet volume was 5 micro-liters. Images of droplets were acquired immediately on contact of the droplet with the coupon surface. Five markers were placed around the droplet image and both right and left angle images were calculated using the VCA OptimaXE software. Ten droplets were measured along the length of each sample. The mean contact angle and the standard deviation were calculated for each sample. Calibration of the VCA Optima instrument was conducted using a PTFE substrate and de-ionized water under ambient conditions (47 % RH, 23 °C). The PTFE surface was cleaned sequentially with acetone, methanol, and distilled water. The cleaned PTFE was preserved in a hot air oven.

2.3.1 Contact Angle Results

Materials with contact angles at or greater than 90° are considered hydrophobic while materials with contact angles less than 90° are considered hydrophilic. The results for the contact angle measurements as a function of humidity for Kapton® polyimide film are given in Table 2 below.

Table 2: Contact angle measurements for Kapton® as a function of relative humidity.

% RH	Contact angle (°)
70	72.25 ± 3.58
50	71.67 ± 3.14
< 10	73.79 ± 3.52

The variation in humidity appears to have little effect on the water contact angle for Kapton® within measured parameters. The standard deviation observed in the experiments may be due

to surface contamination. The results show that the surface is mildly hydrophilic meaning it can adsorb moisture. Once moisture is on the surface, charge carriers become more mobile. If there is enough moisture, the charge can dissipate via a provided ground path in stark contrast to resistance measurements which classify the surface as insulating at all humidities. The lack of correlation between resistivity and charge decay has been observed by several authors [7-9] and was not unexpected.

3.0 Spark Incendivity Testing

The leading technique to evaluate the propensity of a material to be the source of an incendive spark requires the Spark Incendivity Test, a new International Standard used by the International Electrotechnical Commission (IEC) entitled, "Electrostatic Classification of Flexible Intermediate Bulk Containers (FIBCs)" [1]. This test method has been successful for evaluating the safety of FIBCs which are widely used for the storage, transportation and handling of powdered, flaked or granular material throughout industry. The mishandling, improper grounding, and poor electrostatic dissipation properties of FIBCs have led to several hazardous events which have caused injury and/or death to workers in some cases.

The use of the new IEC test standard has greatly decreased the number of incidents by addressing the ESD hazard directly. Essentially the test method is to charge a material either by corona charging and/or triboelectric charging and then purposely discharge the surface of the test material (in the form of a spark) using a metal sphere while in the presence of a flammable gas mixture. If the energy of the spark has at least the energy equal to the minimum ignition energy (MIE) of the gas mixture, then the resulting discharge will be capable of igniting the gas mixture. This test method correctly measures an insulator's propensity to extract sufficient ignitable charge from its surface when a worker and/or a conductive object are nearby.

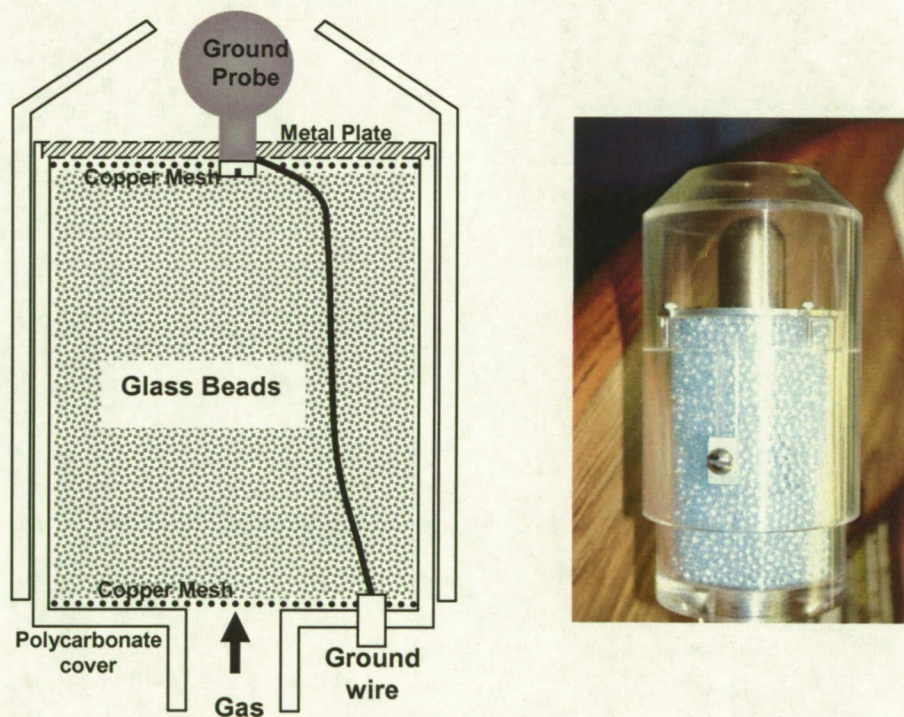


Figure 3. A schematic and picture of the Spark Incendivity Probe. The glass beads help mix the hydrogen and air as well as prevent back propagation of the flame.

To perform this test, a Spark Incendivity Probe (SIP) is used. Gases with a known MIE enter into a polycarbonate mixing chamber full of glass beads to ensure uniform mixing and to serve as a flame arrestor to prevent back propagation. The gas mixture passes first through fine copper mesh and then into the mixing chamber before passing through a second copper mesh and a perforated metal plate. The gases then surround the brass electrode which is electrically grounded. It is here that the ignition occurs.

The proposed test standard normally calls for gas mixtures of 13% ethylene in air, which has an MIE of 0.14 mJ. This is the case for FIBC's in the presence of methanol environments as seen normally in industry. Here however, testing is performed using hydrogen in air at stoichiometric mixtures (30% H₂ and 70% dry air as before) having a much lower MIE of 0.02 mJ to represent the worst-case scenario of a charged material in the presence of a hydrogen-enriched atmosphere. Electrostatic charging occurs in several forms including triboelectric charging from workers handling materials, charged items or equipment, charged liquids and vapors during typical processing and operations. The Spark Incendivity Test using hydrogen-air mixtures can check to see if materials are susceptible to incendive discharges if highly charged in very sensitive environments. If incendiary discharges do not occur the materials could be deemed safe as is and normally do not require further testing.

Samples are typically mounted on an insulating frame using clips. Once mounted, the materials are acclimated for up to 24 hours at both 20% \pm 5% relative humidity and 50% \pm 5% relative humidity. Charge is normally applied to the materials in two ways. The first method to apply charge to the materials is by tribocharging with wool. The wool cloth is repeatedly rubbed against the test materials until they are saturated with charge. The second method is corona charging. A high-voltage power supply (Glassman Series EH) is connected to a corona needle plate to create ions in the air that deposit onto the surface of the test material. The voltage is set to -30kV to deposit charge levels similar to what is seen in FIBCs. After deposition onto the surface, the resulting surface potential is measured using a JCI 140 Fieldmeter (John Chubb Instruments) and recorded.

After the test material is fully charged, the gas mixture is allowed to flow through the probe for at least 30 seconds. The probe (Figure 3) then approaches the charged material with the speed of approach of about (0.75 \pm 0.25) m/s. Too slow an approach would cause corona to reduce local charge levels and too fast an approach would cause quenching of the nascent flame kernel. In the case that the extracted charge is sufficient to cause an ignition of the hydrogen-air mixture, the ignition event is recorded. Ignitions are monitored by listening to the loud pop sound of the hydrogen-air mixture. The violent nature of a hydrogen-air mixture is easily distinguishable from other background noise. In some cases sparks can be observed as charge travels from the sample material to the probe. After charging, the Spark Incendivity probe approaches the sample in five separate locations. Ignitions, non-ignitions and observable sparks are all recorded for every approach.

Experiments were performed at the Space Life Sciences Laboratory at Kennedy Space Center (KSC) within a controlled environment chamber (named OES-2). The large walk-in chamber is capable of maintaining accurate temperature and relative humidity control between 20%

and 90%. All of the controls for the experiments including the power supplies, hydrogen and oxygen gas monitors (Commander), and the MKS cluster controller for the mass flow controllers are kept outside the OES-2 chamber free from the hazardous gases. Mass Flow Controllers operated by the MKS Cluster Controller are used to fix the flow rates of the hydrogen at 0.9 liters/min and air at 2.1 liters/min which provides the stoichiometric mixture (30% H₂ and 70% dry air). A picture of the test setup within the chamber is given in Figure 4.

Two operators are required for Spark Incendivity testing. One operator performs the Spark Incendivity test inside the OES-2 chamber while the other operator is responsible for monitoring hydrogen and oxygen levels, humidity levels, controlling the corona power supply and performing data acquisition. Both operators can communicate via walkie-talkies. Internal cameras can supply live action images through the web as well. There are five probe approaches for each trial. The locations are generally the top-left, top-right, middle, bottom-left and bottom right positions of the test material. Ignitions per total number of trials are recorded and the results are tabulated in Table 3 below for triboelectric charging with wool.

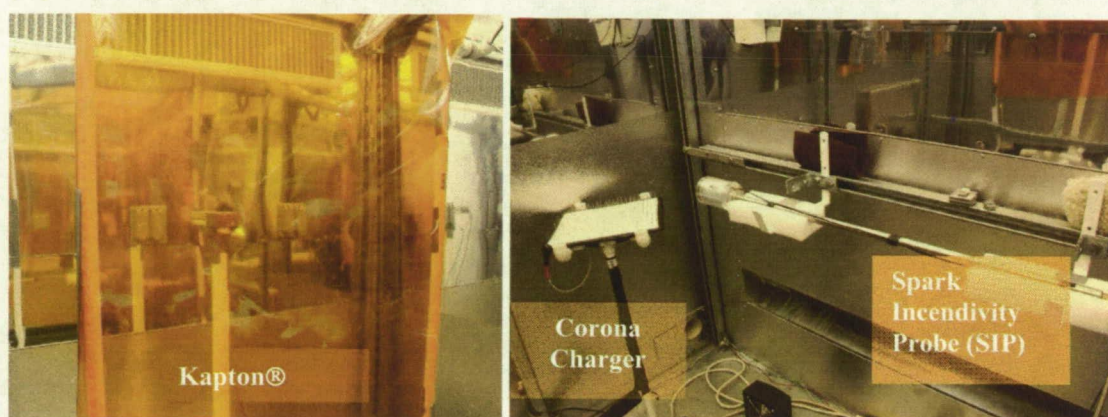


Figure 4. (Left) Kapton® mounted within the OES-2 Chamber. (Right) The Spark Incendivity Probe and the corona charger used to supply surface charge to test materials.

3.1 Spark Incendivity Results

Table 3. The summary of Spark Incendivity Results tribocharged with wool.

50% RH		20% RH	
Surface Potential	# of Ignitions/trial	Surface Potential	# of Ignitions/trial
> -20 kV	2/50	> -16 kV	1/25

The surface potential measurements during the Spark Incendivity tests were performed by manually holding the JCI 140 fieldmeter at the 10 cm calibrated distance from the highly charged polyimide surface, per normal operating procedures. In nearly all cases the surface potential readings exceeded the voltage limit capabilities of the meter. Only the initial rub during the 20% RH test was the voltage below -20 kV according to Table 3. Thus the true surface potential exceeded -20 kV in most cases.

Of the 375 approaches (5 per trial and 75 trials) there were two ignitions at 50% RH and one ignition at 20% RH of highly charged Kapton®. These results indicate that Kapton® is

susceptible to incendive brush discharges and hence **fails** the Spark Incendivity Test in accordance with the standard. Since the material failed the triboelectric charging portion of the test, there was no need to carry out the corona charge portion of the test.

3.2 Brush Discharge Onset Testing

Materials that fail the Spark Incendivity test standard is enough to rule them out for use in Division I areas (defined by NFPA 70 [10]) which are those likely to encounter flammable gases most of the time. Insulator materials will fail the Spark Incendivity test standard quite often for several reasons. First the material will acquire significant charge when rubbed vigorously with an excellent insulator such as wool. Furthermore, the approach of a well grounded conductor of ideal geometry ensures that the maximum amount of charge will be extracted during the sparking event. Thus the Spark Incendivity test standard provides a very conservative approach to material selection in highly susceptible areas by essentially ruling out the use of most insulators. However, should the same criterion hold for materials in Division II areas or those that will not likely encounter flammable gases at all times? The use of insulator materials is highly desirable in many operations due to ease of operations, reduced cost or other necessary requirements in which the use of good conductors or statically dissipative materials is simply not possible. The test may not be ideally suited for less stringent environments which are more commonplace throughout industry. For example, the voltage levels attained with wool rubs may not be representative of those typically generated during operational use. Therefore, it would be more desirable to check if the charge generated during typical operations can bring about an incendive brush discharge rather than one generated by an unlikely extreme charging scenario.

The new procedure developed to evaluate a material in its operational environment is as follows. First we determine the surface potential at which onset of incendive brush discharges occurs. This requires applying different amounts of charge to the material's surface and checking whether the charge applied is sufficient to cause an ignition of the spark incendivity probe. Next we determine whether the materials that are expected to tribocharge Kapton® during operational use can provide the surface potentials necessary to cause incendive brush discharges. This is determined by two methods. The first is to simply measure the surface potentials before, during and after tribocharging and the second is to perform the tribocharging event while immersed within the flammable gas atmosphere of known MIE.

To find the surface potential required for an incendive brush discharge to occur, we performed several Spark Incendivity tests while accurately monitoring the surface potential before, during and after probe approaches a step not performed during Spark Incendivity testing. This required the JCI 140 fieldmeter to be present and stationary at all times and have ability to "see through" the material. To do this we took advantage of the dielectric properties of the polyimide in which the electric field on one side easily transmits through the material on the other side. Placing the JCI 140 at 10 mm away on the back side of the Kapton® allows constant monitoring. The only problem is that the large surface potentials generated always exceeded the meter's capabilities which went out of range well above -20 kV. Thus to get an

accurate reading of the surface potentials required a new and lengthy calibration process for the JCI 140.

3.2.1 Calibration

The JCI 140 is designed to measure surface potentials at 10 cm away from a source but because measurements exceeded the range, we decided to move the JCI 140 further away from the source and measure a correction factor which can adjust the data later. This was performed by placing a large metal plate of the same size as the polyimide sheet at different distances from the JCI 140. A 2'x3' aluminum covered board was placed at 10 cm, 20 cm and 30 cm away from the JCI 140 mounted on an insulated post. (The JCI 140 on top of the post and behind the polyimide sheet can be seen in the left graphic of Figure 4). Voltages were applied to the aluminum sheet using a Glassman HV power supply. The resulting calibration showed that simple multiplication rules applied to the measured JCI potentials as a function of distance away. For example the correction factor was 1 at 10 cm away which is the standard measurement distance (1:1). The correction factor was 1.5 at 20 cm away or 3:2 and the factor was 2 to 1 at 30 cm away. Thus a measurement of 10 kV at 30 cm away corresponded to a real voltage of 20 kV. Thus the new scale factor allowed the JCI to be placed much further away from the Kapton® making it possible to record much higher potentials.

Although this technique should apply well for a large flat metal plate, it should not be assumed that fields emanating from an insulating surface behave in a similar manner. Voltages applied to metal plates produce fields that may not necessarily correlate to electric fields generated by charges on the surface of an insulator. The electric field lines emanating from a conductor must be uniform and perpendicular to the surface (as a consequence of mobile charge carriers) whereas in insulators they need not be; there could be oppositely charged or uncharged patches whose field lines diverge in all directions. For example, one might imagine that as the fieldmeter is moved away, electric fields from oppositely charged patches become observable which lowers the overall measured potential.

In order to see if the same scale factor rules discovered above applied for a charged sheet of Kapton®, the following tests were performed. First, charge was applied using at least two types of wool and PTFE felt to make sure that different charges gave similar results. Next, the surface potential was monitored at 10 cm away as a result of the rub. After it was recorded, the JCI 140 was slid back to 30 cm and the resulting potential measured. The results are shown in Figure 5.

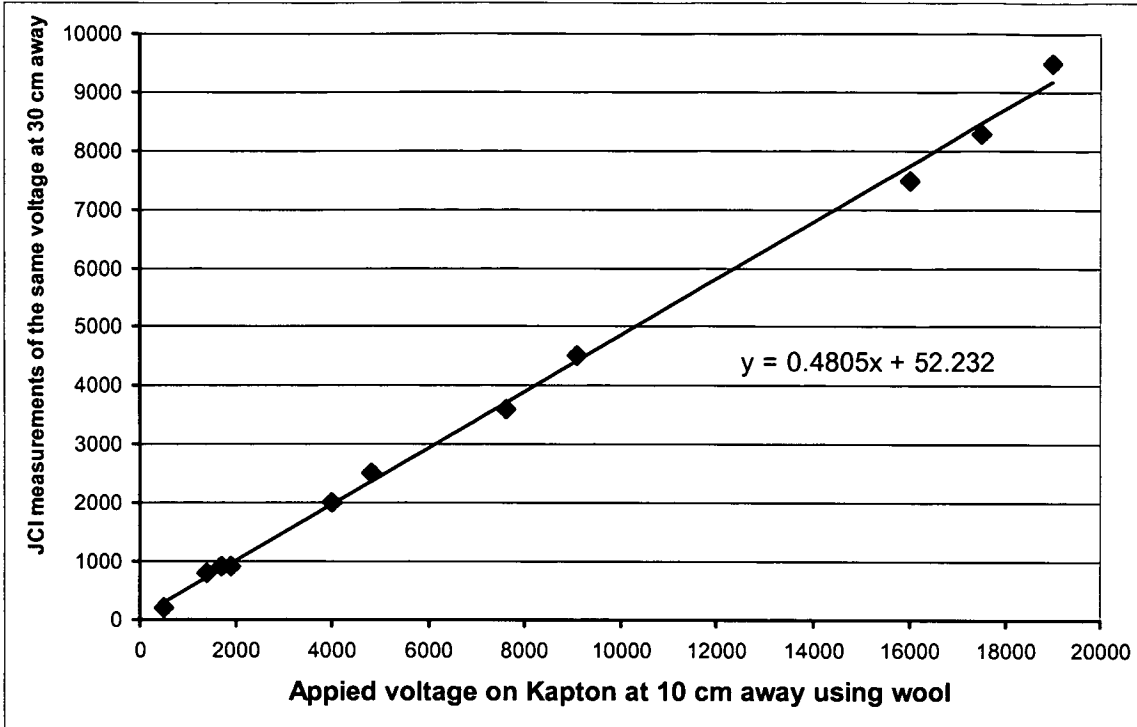


Figure 5. The measured voltage on Kapton® at 30 cm as a function of measurements at 10 cm. Charge was applied using three separate materials. The slope of the line indicates the same 2:1 ratio for the correction factor as for metal calibration plates.

Figure 5 shows that regardless of charging material, the slight spatial changes of the surface charge density (which is expected on the surface of an insulator) did not affect the overall surface potential measured using the JCI 140 fieldmeter. The correction factor remains unchanged which means the same number of field lines that enter the JCI 140 fieldmeter at 10 cm also reaches the meter at 30 cm away which is identical to the metal case. Thus the correction factor for a charged sheet of Kapton® matches those of a large metal plate. It should not be generalized for other insulators tribocharge using different materials. A calibration of this type has never been reported in the literature as far as we know.

It was also observed that in some cases the placement of the JCI 140 at the greatest distance away from the polyimide (30 cm) was still not sufficient to keep the measurements within the range of the fieldmeter signifying potentials exceeding -60 kV. Although the measurements were outside of the operating range of the device, the JCI 140 still record potentials above the -20 kV range which can be monitored through the device's analogue output. However, it was unclear at the time whether these readings were indicative of true values since the manufacturer can not guarantee readings above -20 kV. As a result another test was performed using the JCI 140 to ensure that potentials above this range were accurate. This test used a high voltage plate connected to a Glassman HV power supply capable of reaching extreme potentials up to ± 60 kV. If the JCI 140 measured these potentials accurately, then measurements above the operating range could be trusted.

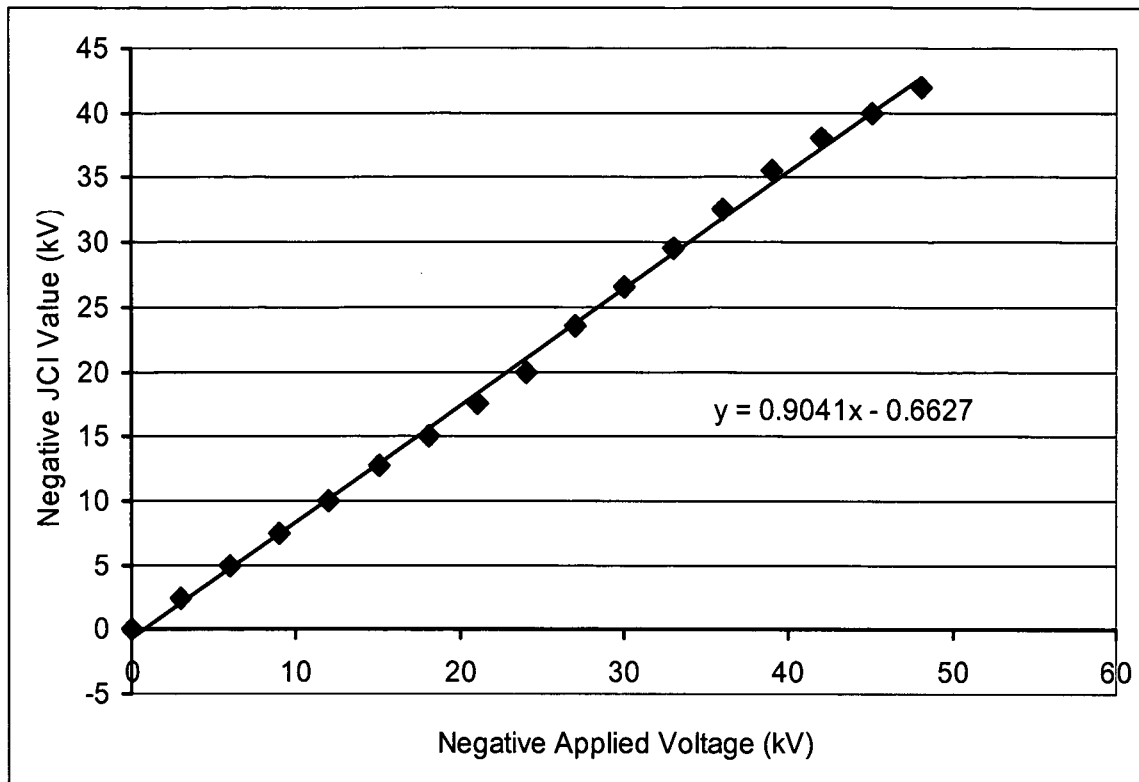


Figure 6. The negative values of the measured voltage a metal plate 10 cm from the JCI 140 for calibration above -20 kV. The slope of the line is linear giving a simple correction factor.

When performing tests of this nature certain precautions were necessary. One such precaution was to perform the entire test within a large Plexiglas box to avoid shocks to personnel. Another precaution taken was to cover the edges of the metal plate with electrical tape which prevented corona from sharp edges as well as sparks and/or arcing to the JCI 140 as well. The high voltage output of the Glassman HV power supply was checked using both the voltage monitor on the back of the instrument and verified using a HV reducer probe connected to a calibrated Fluke 87 V multimeter (cal # M86236).

The results given in Figure 6 show that the linear behavior of voltage applied to voltage measured is an excellent indicator that the device is working properly above its specified range. There is only a slight correction factor of 1.11 that needs to account for the offset. One interesting feature to note is that although the JCI 140 can measure negative voltages above -20 kV, it can not measure positive voltages above +20 kV. Above this range the meter and the analogue output give null results. Fortunately for the materials studied here, Kapton® charges negatively consistently when rubbed with wool.

3.2.2 Results

With confidence that accurate surface potential measurements can now be performed to record the onset of brush discharges, testing commenced in a similar manner to Spark Incendivity Testing reported earlier. The only difference was that there were more accurate measurements taken of Kapton's® surface potential. The JCI 140 was placed 30 cm from the back of the polyimide mounted in an identical fashion of Figure 4. It was decided to perform

this testing at higher relative humidities since it has been known for some time that more incendive discharges occur at this humidity [9]. The reason is simply that charge is more mobile on the surface and thus more charge can contribute to the discharge. When this happens, incendive sparks are more likely to occur.

The polyimide's surface was rubbed with wool and the Spark Incendivity Probe was used to test the incendive brush discharge onset. Early tests to measure the onset of brush discharges were unsuccessful at 70% relative humidity. Both the wool and the Kapton® were too moist for significant charge deposition. Successful tests were only obtained at 50% relative humidity. The corrected values for the onset of brush discharges are shown in Figure 7 and Figure 8. These tests were repeated dozens of times and only a few are presented in each graph.

Each amount of rubbing with wool deposits a certain amount of charge until the surface saturates. For Kapton® the saturation occurs at about -85 ± 5 kV. Three ignitions were recorded during the test run of Figure 7 which are highlighted by the arrows. These ignitions occurred at the surface potentials of -67 kV, -79 kV and -84 kV. None of the following tests contained ignitions that occurred at lower potentials than these. The first ignition, which was the lowest potential measured, caused the surface voltage to drop by 42 kV from -67 kV down to -25 kV as a consequence of the large amount of charge displaced during the discharge.

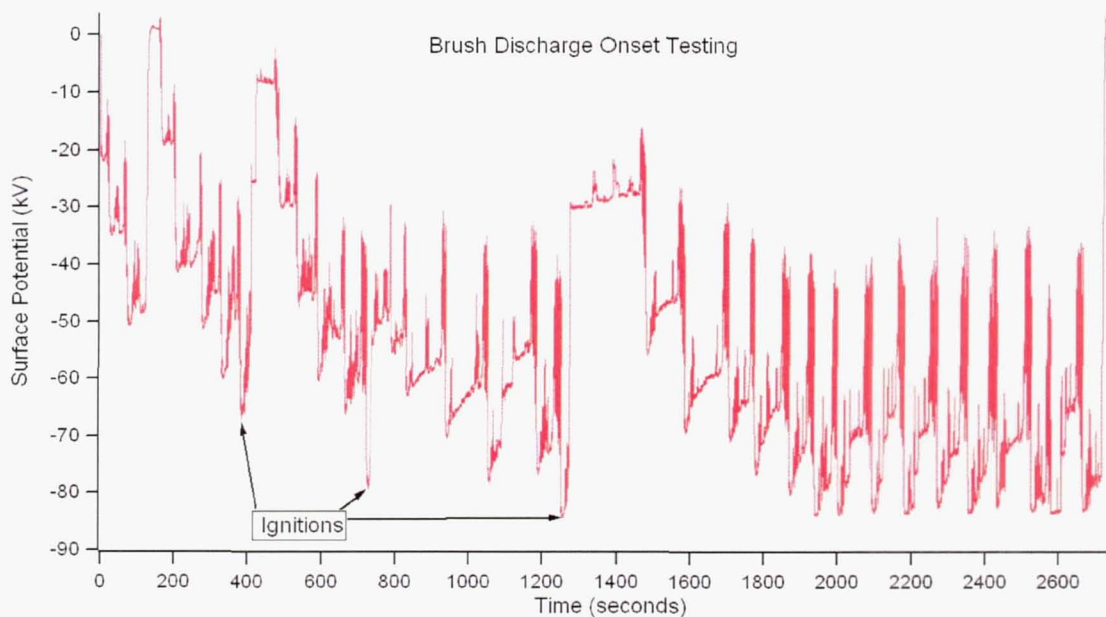


Figure 7. The surface potential measurements during the onset of incendive brush testing. Several dozen tests were performed (only a few are shown) at 50% relative humidity.

A close-up of data in Figure 8 shows how the Spark Incendivity Probe and the test conductor affect the measured surface potential. Rubbing can also be easily detected. This graph is included to highlight the accurate nature of the JCI 140 fieldmeter reading and also serves to validate the use of the technique.

The final results indicate that incendive brush discharges can only be expected to occur if the potential for Kapton® HN is at or exceeds -67 kV which is a remarkably high value. Typical

charged-insulators are thought to become incensive at about 20-25 kV according to the literature [11].

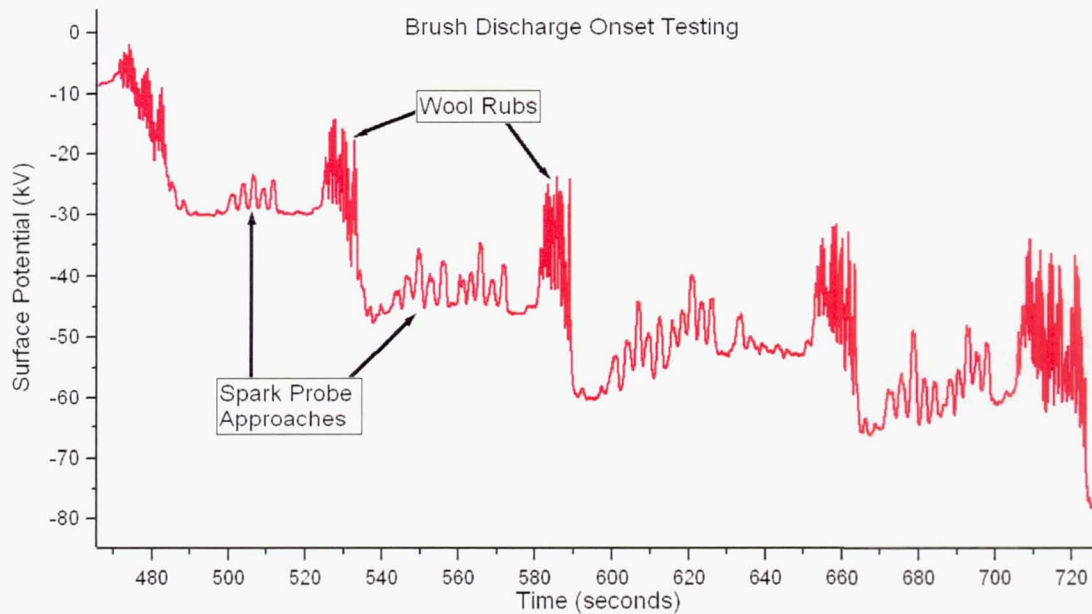


Figure 8. A close-up of the surface potential measurements during the onset of incensive brush testing.

4.0 Representative Testing

Once the onset of incensive brush discharges is known, the next step is to check if the voltage levels required can be generated by the materials used in the relevant environment. As an example, three candidate materials that might tribocharge Kapton® for a particular operation include: well-grounded metal (titanium) and two insulating coatings - epoxy (Koropon) and black paint on an aluminum plate. The resistive properties of the rubbing materials will be classified followed by surface potential measurements generated by tribocharging.

4.1 Resistivity tests

A Koropon-coated sample of aluminum shaped like a paint brush roller was provided for ease of rubbing against the polyimide. The piece has overall dimensions of approximately 7.5" × 7.5" with a large flat side with rounded edges with dimensions of 7.5" × 4.5". In addition to the paint-brush shaped Koropon sample, there were two flat aluminum plates one coated with Koropon epoxy and one with black paint provided for testing. The thickness of Koropon coating is between 0.006" - 0.009" and the black paint is 0.0018" - 0.0022" thick. Both coatings are deposited onto a 1 sq ft 1/16" thick aluminum plate. The uncoated titanium sample is 1" thick with dimensions of 7" × 11" that weighs over three pounds. Black electrical tape was used to prevent all metal samples from generating corona at high electrostatic fields. Pictures of each of the materials are shown in Figure 9. The surface resistances and volume resistivities of the test materials are given in Table 4. Both the Koropon and the black-paint samples are highly resistive and behave as insulators while the titanium sample is a good conductor.

Table 4. Surface and Volume Resistivity of test materials.

	Surface Resistance	Volume Resistivity
Koropon (0% RH)	$351.67 \pm 77.31 \text{ G}\Omega$	$673.7 \pm 134.77 \text{ T}\Omega \text{ cm}$
Black Paint (0% RH)	$429.83 \pm 126.46 \text{ G}\Omega$	$671.66 \pm 466.44 \text{ T}\Omega \text{ cm}$
Titanium (0% RH)	$173.12 \pm 76.36 \text{ }\Omega$	- $\Omega \text{ cm}$

(Note: M Ω is mega ohms $10^6 \text{ }\Omega$, G Ω is giga ohms $10^9 \text{ }\Omega$, and T Ω is tera ohms $10^{12} \text{ }\Omega$)

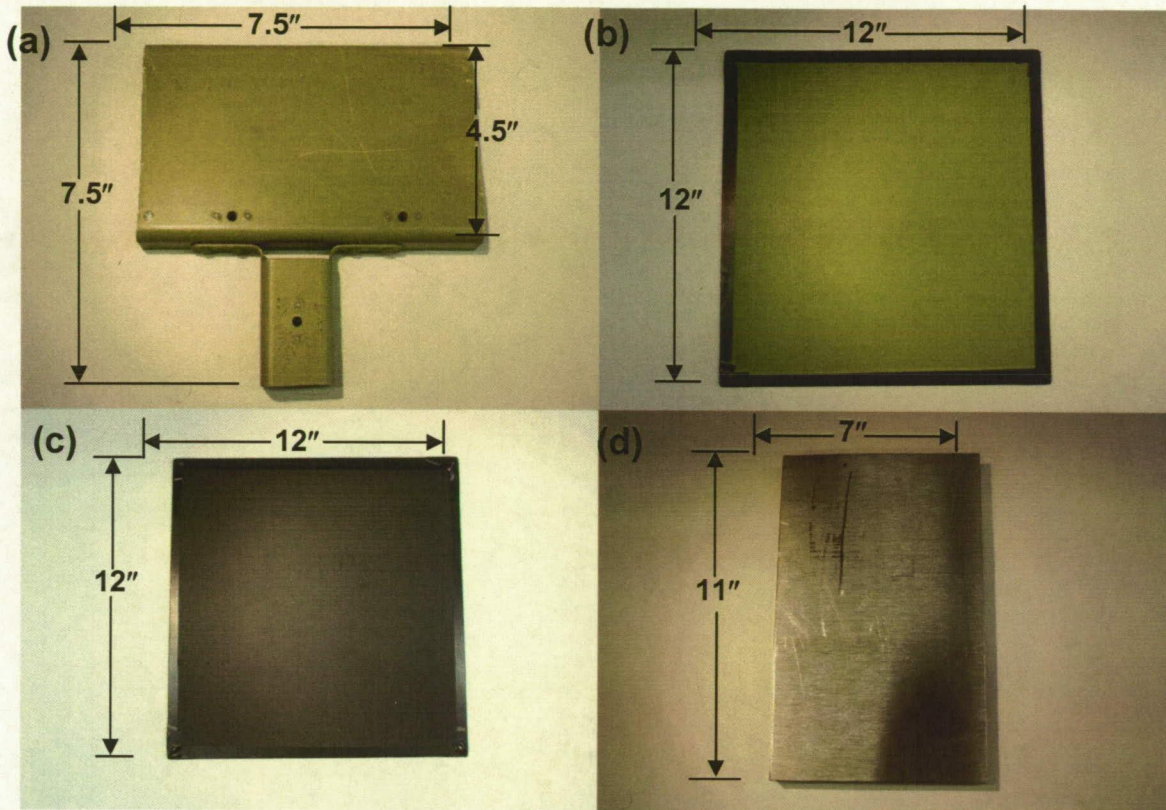


Figure 9. (a) The paint-brush shaped Koropon coated aluminum sample. (b) Koropon coated aluminum plate sample. (c) Black paint coated aluminum plate sample. (d) Titanium sample.

4.2 Triboelectric and Spark Incendivity tests

Early tests focused on the chargeability of the Kapton® using the paint-brush shaped Koropon sample because of the ease of use. Tests were performed on the large 3' x 3' Kapton® sample mounted within the OES chamber as before acclimated at 30% relative humidity. The JCI 140 was mounted behind the polyimide as in previous tests. Rubbing was performed by hand and the surface was discharged using an air ionizer between runs. The surface potentials of a dozen rubs using the “paint-brush” shaped Koropon sample is given in Figure 10.

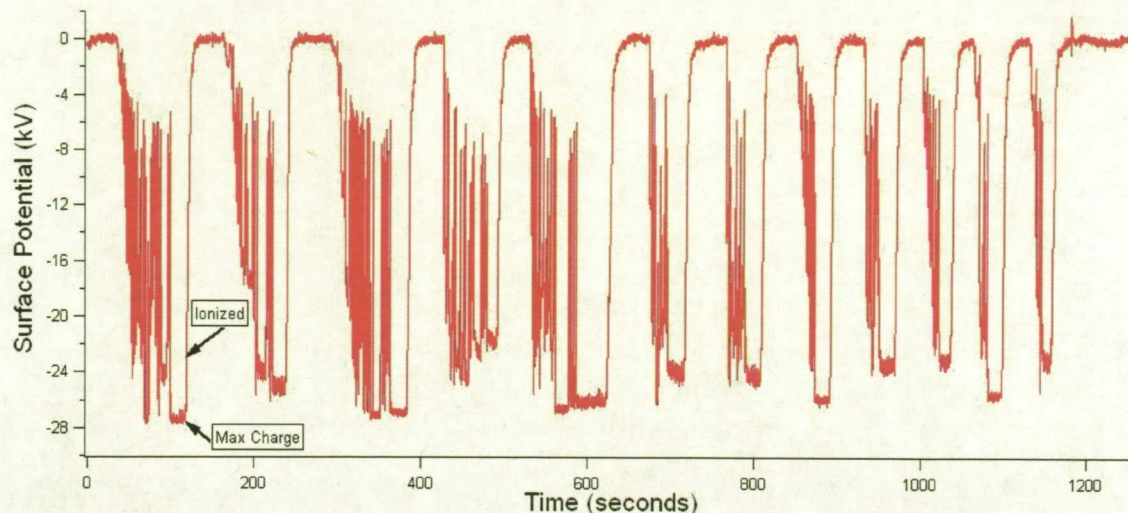


Figure 10. The surface potentials generated using the paint-brush shaped Koropon sample at 30% RH.

Figure 10 shows that rubbing with the Koropon sample deposits a consistent amount of charge with average surface potentials of -25.35 ± 1.6 kV. Although these levels are well below the -67 kV as measured by the onset of incandive brush discharges of the previous section, the literature states that potentials on the order of -25 kV should produce brush discharges [11]. Therefore, additional Spark Incandivity tests were performed to be sure.

Spark Incandivity tests were performed at 20% RH inside the OES chamber using the paint brush Koropon-coated sample as the charging material similar to Figure 10. The average charge levels were -23.86 ± 3.16 kV and there were no ignitions for all 50 tests. Thus the Koropon sample was unable to generate surface potentials necessary for incandive brush discharges in agreement with the -67 kV requirement discovered for the onset of incandivity reported earlier.

To perform Triboelectric charging tests of the three other sample materials, a new geometry for the Kapton® had been devised. The polyimide was cut and stretched over soft foam mounted to a more rigid blue foam. The soft foam serves to absorb the impact as the Kapton® rubs as it rubs against the test material to ensure excellent contact. The hard foam provided rigidity for both the Kapton® and soft foam. Foam was chosen not only because it is lightweight and rigid but it is also a good dielectric. The electric fields emanating from the charged Kapton® pass through the foam with minimal distortion of the electric fields. Two sample holders were made using a grey and white soft foam backing. The soft foam backing was slightly smaller than the larger hard foam to minimize Kapton's ® contact with the electrical tape on the Koropon and black-painted samples. Each system shown in Figure 11 was used to rub against the new heavier tests materials.

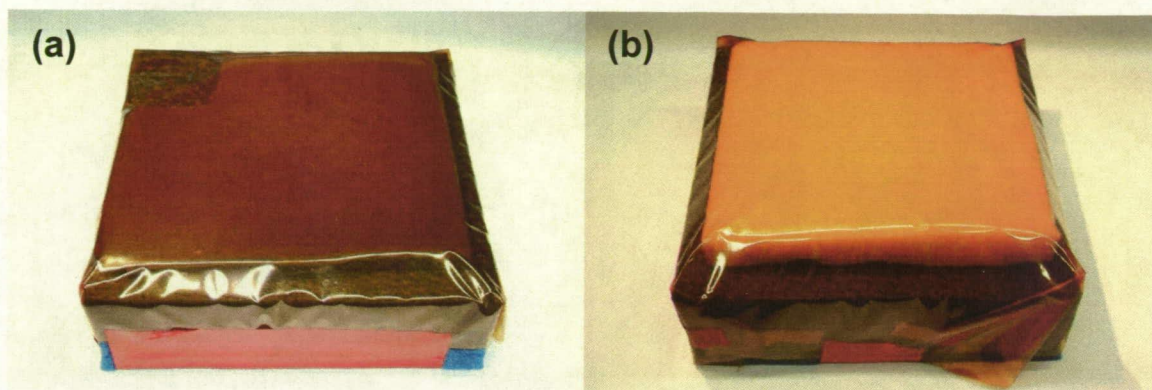


Figure 11. A picture of a portable and lightweight Kapton® holder using (a) grey foam and (b) white foam. The holders have dimensions of 10"×10"×4".

The geometry of the new holders vastly improved testing capabilities. The first tests with this new system were to simply rub the surface of the Kapton® using the paint-brush shaped Koropon sample. After 3 rubs, the surface potential of each foam holder reached a maximum surface potential of -25.24 kV measured with the JCI 140. These values compared well with those using the paint-brush sample rubbed against the large mounted sheet of Kapton® inside the OES chamber thereby confirming that the geometry of the new holders can generate accurate and representative surface charges on the polyimide.

The other three flat samples were allowed to acclimate at 0% RH for several days prior to testing. Testing was performed inside an ETS Environmental Chamber by simply rubbing the Kapton® against the grounded aluminum and titanium samples and immediately placing the holders in front of the JCI 140. The force of rubbing or the pressure applied did little to change the magnitude of the surface potential measured for each test. The maximum surface potential generated on the Kapton® when rubbed with Koropon was -46.63 kV for 141 rubs; for the black-paint sample it was -46.0 kV for 127 rubs and for the titanium sample it was -45.02 kV for 81 rubs. All three materials are capable of depositing nearly equal amounts of charge to the polyimide. The different amounts of surface charge applied to Kapton® using two samples of Koropon could be explained by the fact that the paint-brush shaped sample was 20+ years older than the freshly coated aluminum plate sample. Although the values were different, each sample deposited a consistent amount of charge to the polyimide.

It is a common misconception that well grounded metals cannot tribocharge an insulator. However, the uncoated grounded metal used here is capable of depositing large amounts of charge onto the polyimide surface.

Another series of tests were performed to see how the Kapton® charges as a result of contact with itself. These tests were done by simply rubbing the Kapton® grey holder against the white holder. In all cases there was electrostatic charging of the Kapton® which ranged from -19 kV to + 7 kV. The charging of similar materials is well known but not well understood in the scientific community. No further discussion on this topic is presented.

4.3 New Sample Holder

Spark Incendivity tests were performed since it was important to verify that these charge levels were not capable of generating incendive brush discharges as before because of the relatively high surface potentials. In order to take advantage of the thin dielectric and measure the surface potential through the back of the polyimide as before, a new sample holder was built. The requirement to keep the JCI 140 at least 30 cm away from Kapton® was no longer necessary because of the smaller potentials generated by the test materials allowed a new design which placed the fieldmeter to sit only 10 cm away. The JCI 140 could now be mounted within a cutout in the center of the foam and measurements can be made before, during and after separation of the test material. The entire ensemble shown in Figure 12 can be used manually for triboelectric charging since it is now lightweight and portable. The ability to measure the surface potentials of a material accurately *during* both the charge process and separation has seldom been performed. Such techniques developed here are not presently available with commercial instrumentation.

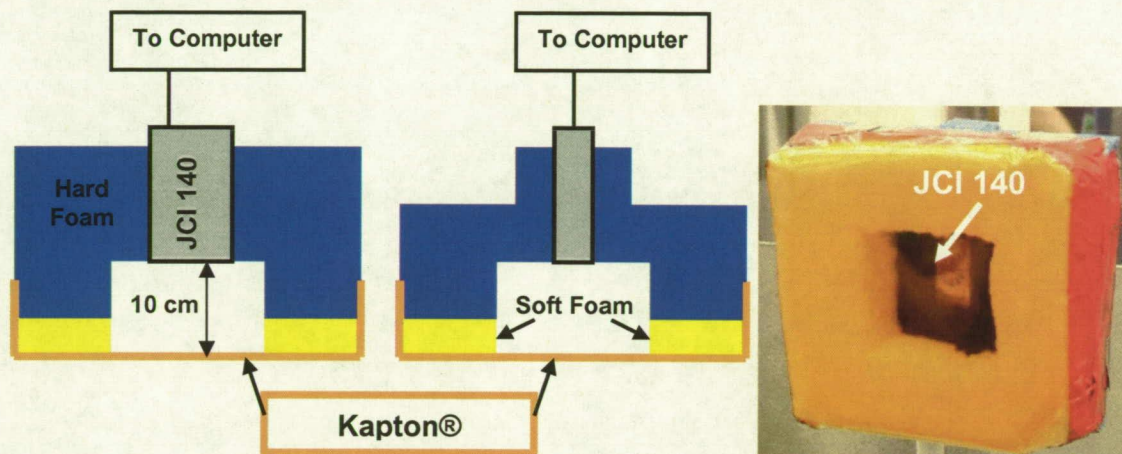


Figure 12. A schematic (front and side view) and picture of a more advanced Kapton® holder that contains an embedded JCI 140 Fieldmeter 10 cm behind the front of the charged material.

The first test using the new Kapton® holder was Spark Incendivity testing. These tests were performed by rubbing the 12" square samples of Koropon and black paint-coated aluminum up against the Kapton® holder firmly mounted inside the OES chamber in the vertical position. The Kapton® had to be mounted vertically in this case to allow the user to approach the charged surface with the Spark Incendivity Probe. Each flat plate sample was properly grounded and fitted with a plastic holder mounted on the back for manual rubbing. The surface potential monitored throughout these tests are shown in **Error! Reference source not found.** for Kapton® rubbed with the Koropon epoxy coated aluminum plate.

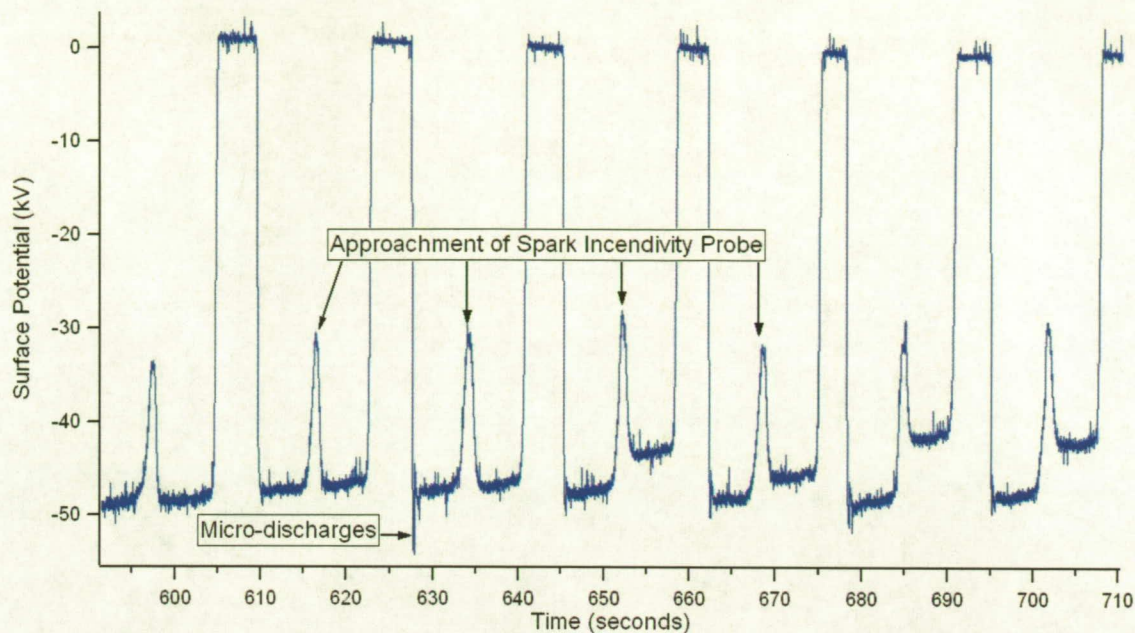


Figure 13. Kapton's® surface potential measured during Spark Incendivity Testing when charged with Koropon coated aluminum.

As expected, as the Koropon sample approached the Kapton® the surface potential read zero when oppositely charged surfaces recombine. Upon separating the materials from one another, the resulting potentials were about ~ 50 kV as seen in **Error! Reference source not found.** At times during the separation small discharges called micro-discharges, which are well above the noise of the instrument, were observed with the JCI 140 indicating that large amounts of charge was deposited onto the surface of the Kapton® causing electric breakdown of the air which occurs when the electric fields exceed 3×10^6 V/m. The overall magnitude of the surface potential as measured by the JCI 140 immediately upon separation is certainly lower than the actual potential difference between the two materials which confines most of the electric field lines. It is not clear whether the amount of charge can generate discharges that are incensive. This phenomena probably occurs quite often when materials are rubbed and then separated but rarely is it measured. Normally one measures the final resulting surface potential which is ~ 50 kV here. The largest potential recorded was -54.8 kV for the Koropon and -47.31 kV for the black-painted aluminum sample.

A few seconds after charge is deposited, the Spark Incendivity Probe approached the surface in an attempt to extract enough charge to cause an incensive brush discharge. There are 7 approaches shown in **Error! Reference source not found.** out of 50 tests performed. Evidence for charge released is found by simply monitoring the surface potentials before and after probe's approach. It can be seen from **Error! Reference source not found.** that tests performed at times 650, 685 and 700 seconds, that the surface potential failed to return to its prior value after the probe made contact. This is because small amounts of charge were transferred to the Spark Incendivity Probe. The highest recorded potential difference before and after probe approaches was -7 kV for the black-painted sample and -14.35 kV for the Koropon sample. These values paled in comparison to those of the brush discharge onset testing which recorded voltage drops as large as -42 kV (Figure 7). As a result, in all 100 tests

there were no ignitions detected using both the Koropon and black-painted aluminum samples which again reinforces the need for potentials at or above -67 kV for Kapton®. Although the Spark Incendivity tests were successful in proving that incendive brush discharges can not occur with the surface charge remaining on Kapton® long after separation, the tests can not rule out the incendivity that may or may not occur during separation.

4.4 Immersion Tests

The final series of tests performed on the sample materials was immersion tests. These tests were designed to see if the micro-discharges that occur when materials separate after electrostatic charging are incendive. Tests of this nature are performed by charging and separating two materials while in the presence of a flammable atmosphere. As the materials separate, micro-discharges occur if the electric field strength exceeds the dielectric breakdown strength of the gas medium. Since it is not known whether these discharges have energies greater than 20 μJ which can ignite stoichiometric mixtures of hydrogen in air, tests performed within the presence of this gas mixture serve as the only method to determine the electrostatic energy released. The only precedent for representative testing of this type that we know of were those performed by us within the past few years [12-15].

A final modification to the Kapton® holder with the embedded JCI 140 was made to prepare for immersion testing. A large Kapton® skirt was attached to the front perimeter of the holder and served to contain the flammable gas mixture before, during and after charging. A picture of this addition is given in Figure 14(a).

To set up for immersion testing, the black-painted aluminum sample or the Koropon sample was mounted horizontally on a frame within the OES chamber. Once mounted, the Spark Incendivity Probe was placed near the edge of the sample to supply the hydrogen-air gas mixture as shown in Figure 14(b). The Kapton® holder with the skirt can now be placed over the assembly as in Figure 14(c) and (d). It is in this configuration that immersion tests were performed by rubbing or pressing the Kapton® holder against the aluminum samples while the gas flowed. The JCI 140 continuously recorded the surface potential throughout the rubs. The holder was lifted at least 12" above the black-paint or Koropon after each rub to ensure that the electric field from the metal samples did not affect the field of the Kapton®.

The potentially large amounts of hydrogen in air forced special precautions to be necessary for the test conductor which included the use of a face shield, heavy apron, large gloves and ear muffs for hearing protection. There was no danger of hydrogen buildup within the entire volume of the OES chamber but the volume between the Kapton® skirt and the metal test samples undoubtedly contained larger amounts of concentrated hydrogen than what is typically seen during Spark Incendivity Testing.

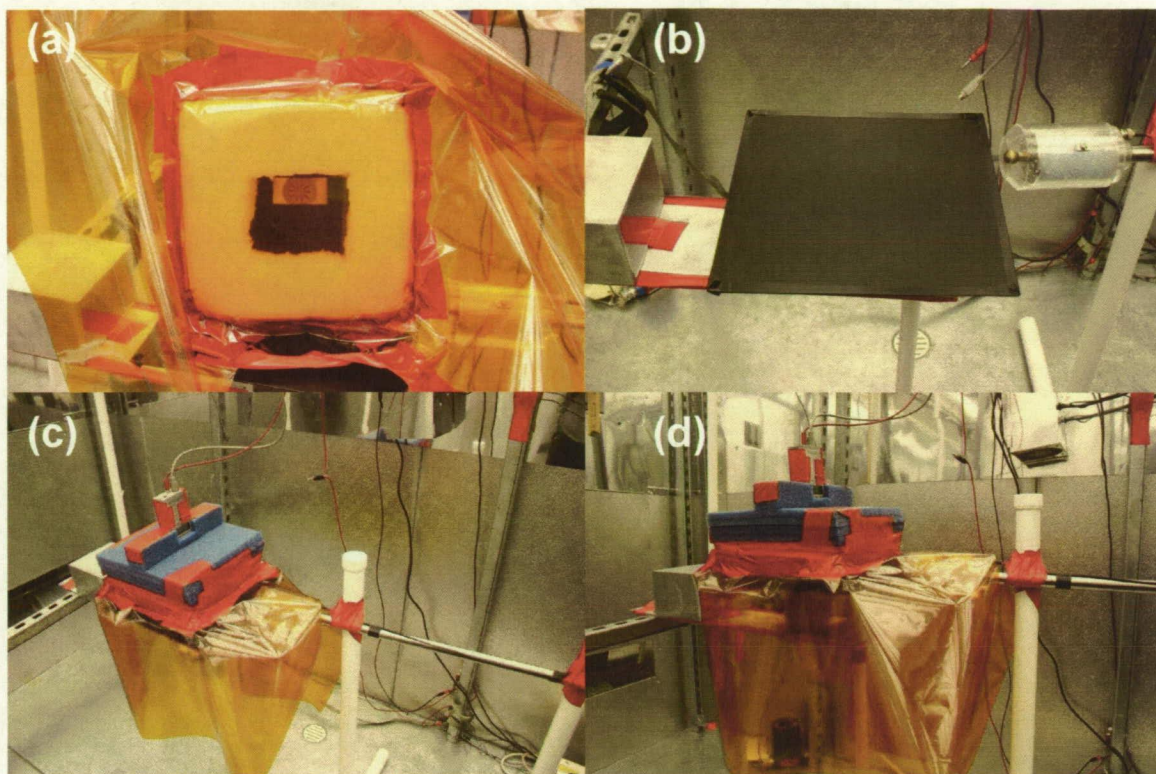


Figure 14. (a) The embedded Kapton® holder with the addition of the Kapton® skirt used to enclose gases. (b) The black-painted aluminum sample mounted in the OES chamber and the Spark Incendivity Probe used to supply H₂-air mixtures. (c) and (d) The final configuration for immersion testing.

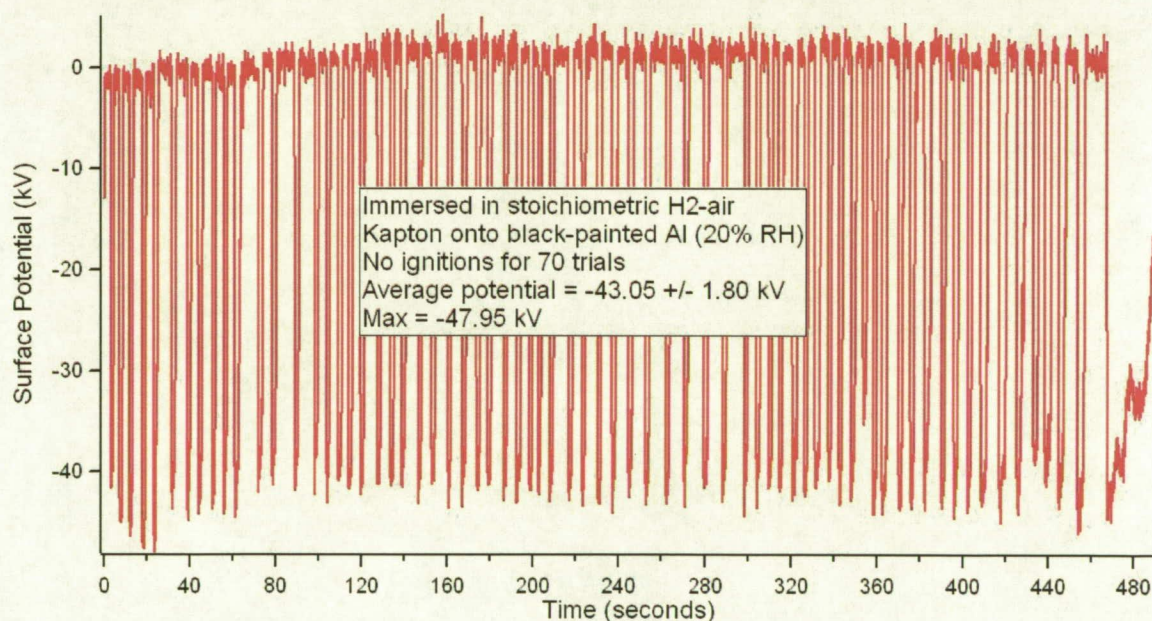


Figure 15. The surface potential measured during the immersed Kapton® tests when rubbed with the black-painted aluminum sample.

Several tests were performed per sample at 20% relative humidity within the OES chamber. There were 70 trials of Kapton® rubbing against the black-painted aluminum sample as

shown in Figure 15 and there were a combined total of 141 total tests taken rubbing the Kapton® against the Koropon-coated aluminum sample as shown in Figure 16 and Figure 17.

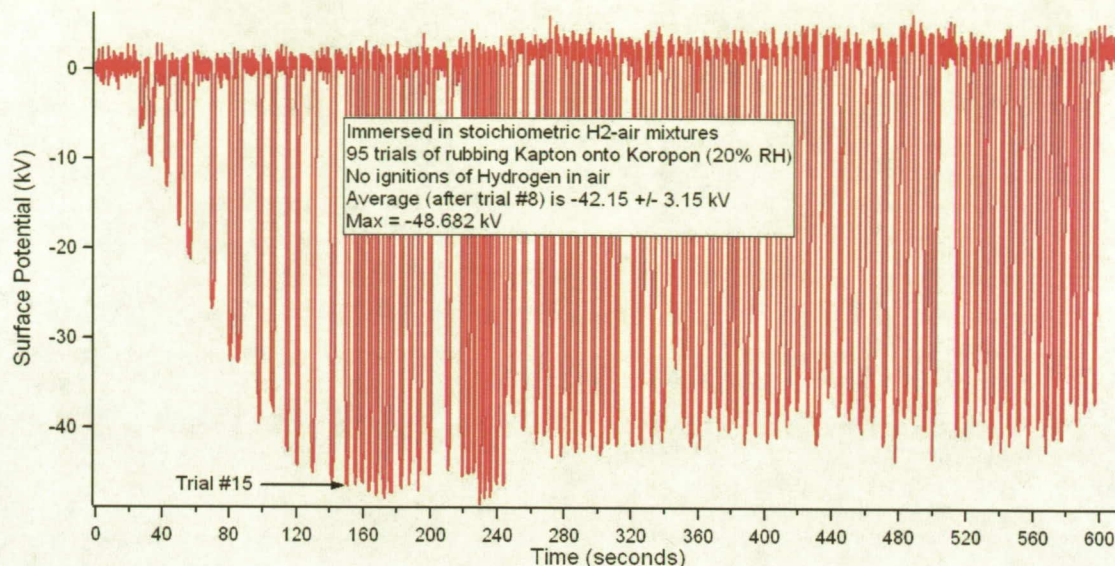


Figure 16. The surface potential measured during the immersed Kapton® tests when rubbed with the Koropon-coated aluminum sample.

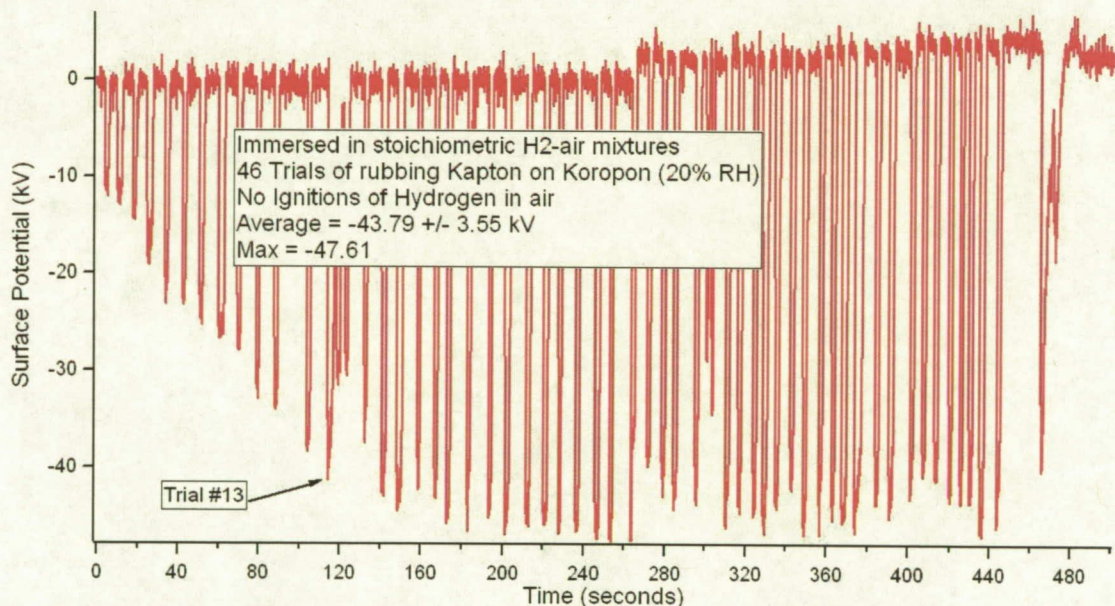


Figure 17. A second set of surface potential measurements during the immersed Kapton® tests when rubbed with the Koropon-coated aluminum sample.

The Kapton® was fully charged immediately upon rubbing against the black-painted sample while several initial rubs were needed for charge saturation for the Koropon coated sample. After saturation, additional rubs were unsuccessful in depositing more charge. The maximum surface potential reading for the black-painted aluminum was -47.95 kV while the maximum potential for the Koropon sample was -48.68 kV. The average values are about -43 kV for both materials.

In all 211 immersion tests using both Koropon and black-paint coated aluminum, there were no ignitions of the hydrogen-air mixtures. This fact combined with the lack of ignitions during the 150 Spark Incendivity tests (including the paint-brush shaped Koropon sample) reinforces the conclusions of the brush discharge onset testing that stated that only surface potentials at or exceeding -67 kV are necessary for incendive brush discharges to occur for Kapton®.

Since the charging levels were the same for titanium as the Koropon and black-painted aluminum, we did not test ignition hazards of Kapton® charged with Titanium using either Spark Incendivity or immersion testing. A summary of the results is given in Table 5.

Table 5. Summary of Results of Sample Materials used to Charge Kapton®

Sample	Total # of Trials	# of Trials in H ₂ /air mixtures	Highest Recorded Surface Potential (kV)	Total # of Ignitions
Paint-brush shaped Koropon	65	50	-25.24	0
Koropon epoxy	332	191	-54.8	0
Black-painted aluminum	247	120	-47.95	0
Titanium	81	0	-45.02	-

5.0 Conclusions

Kapton® by DuPont™ is a highly insulating dielectric with the ability to acquire significant charge upon contact and separation with various materials. The purpose of this paper was to investigate whether it were possible to find surface potential values necessary for incendive brush discharges to occur and to see whether they can be generated in a relevant environment using representative materials expected to come in contact with the material during typical operations.

The first series of tests characterized the material and verified its highly insulating and charging nature. Contact angle measurements proved that Kapton® is slightly hydrophilic meaning that charge can dissipate from the surface provided there is a sufficient ground path in the presence of very high humidity.

Spark Incendivity testing was able to show that Kapton® is able to be a source of ignition of hydrogen-air mixtures. Brush discharge onset testing measured the surface potentials necessary to be at or above -67 kV for ignitions to occur. The onset of incendive brush discharges was confirmed by several tests including rubbing with different materials and rubbing with materials of different geometries under different conditions.

Measurements of the surface potential of a test material during a triboelectric event were performed throughout the contact and separation process using a unique sample holder. Three test materials, two insulators and one conductor, were shown to generate surface potentials up to -50 kV on Kapton® which was below the onset of incendive brush discharge level.

The new sample hold allowed measurements of micro-discharges caused by separation of two highly charged insulators to be recorded. The incendivity of micro-discharges was tested for

the first time by performing tests immersed within a highly flammable environment. These discharges were found not be incendive after several hundred tests.

6.0 References

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